CHARACTERISATION OF THERMOPLASTIC MATRIX COMPOSITES (TPC) USING VARIABLE FREQUENCY MICROWAVE (VFM)

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Abstract: In most industrial microwave processing operations, the frequency of the microwave energy launched into the waveguide or cavity containing the sample is usually fixed. This brings with it inherent heating uniformity problems. This paper describes a new technique for microwave processing, known as variable frequency microwave (VFM) processing, which gets rid of the problems brought about by fixed frequency microwave processing. In VFM processing, microwave energy over a range of frequencies is transmitted into the cavity in a short time, e.g. 20μs. It is therefore necessary to find out the best frequency range to process a material. This paper describes the process of finding the best range frequency for microwave processing of five different thermoplastic matrix composites using the VFM facilities. The optimum frequency band for microwave processing of these five materials was in the range of 8 – 12 GHz. This data enables one to join the above five materials using microwave energy at their most favourable conditions.

INTRODUCTION

In conventional microwave processing, microwave energy is launched at a fixed frequency of either 915 MHz or 2.45 GHz or 5.8 GHz or 24.125 GHz into a waveguide or cavity.\textsuperscript{1} Unfortunately fixed frequency operation has inherent heating uniformity problems like thermal runaway and hot spots. A new technique for microwave processing, known as variable frequency microwave (VFM), has been developed to solve the problems brought about by fixed frequency microwave processing. The technique is geared towards advanced
materials processing and chemical synthesis. It offers rapid, uniform and selective heating over a large volume at a high energy coupling efficiency. This is accomplished using preselected bandwidth sweeping around a central frequency employing a tunable source such as a travelling wave tube as the microwave power amplifier. Selective heating of complex samples and industrial scale-up are now viable. Applications are in the areas of curing advanced polymeric encapsulants, rapid processing of flip-chip underfills, materials characterisation, curing profiles for various adhesives, structural bonding of glass to plastic housing. The Industrial Research Institute (IRIS) at Swinburne University of Technology has two VFM generators; they are the Lambda Technologies VW1500 and Microcure 2100 model 250. VW1500 has a band of 6 - 18 GHz at a nominal power of 125 W, whereas Microcure 2100 model 250 has a band range of 2 - 7 GHz operating at a nominal power of 250W. The cavity dimension of VW1500 was 250 mm x 250 mm x 300 mm; while, Microcure 2100 model 250 had a cavity size of 300 mm x 275 mm x 375 mm. This paper describes the best frequency ranges for microwave processing of five types of thermoplastic composite materials. The five thermoplastic composites were 33% by weight random carbon fibre reinforced polystyrene [PS/CF (33%)], and low density polyethylene [LDPE/CF (33%)]; 33% by weight random glass fibre reinforce polystyrene [PS/GF (33%)], low density polyethylene [LDPE/GF (33%)] and nylon 66 [Nylon 66/GF (33%)].

INTERACTION OF VFM FIELDS WITH MATERIALS
When microwave energy of a fixed frequency, e.g., 2.45 GHz, is launched into a waveguide, e.g., WR340, containing a piece of material, some areas of the material have higher electric field strength than the others. The field configuration of the dominant TE$_{10}$ mode within WR340 waveguide is shown in Figure 1.\(^2\) The unevenness of the field is even more serious if the microwave energy is launched into a multimode cavity because many resonant modes are established. Those areas with higher electric field strength are heated more, creating hot spots, which lead to thermal runaway because material dielectric loss increases with temperature. With variable frequency microwave heating, more than one thousand frequencies are launched into the cavity sequentially over a relatively short period of time, e.g., 20\(\mu\)s. Each incident frequency sets up its own electric field pattern across any cross section of the joint of the test pieces, and therefore results in hot spots at different locations at different time. The modes superimpose with respect to time, effectively creating a time-averaged uniform electric field. When a sufficient bandwidth is used, every element of the test piece will experience hot spots at one or more frequencies during sweeping. The wider the bandwidth, the better will be the uniform heating. Thus, time-averaged uniform heating can be achieved with proper adjustment of the frequency sweep rate and sweep range. Another advantage of the VFM heating is the capability of providing precise frequency tuning to optimise the coupling efficiency.

**CHARACTERISATION OF TPC MATERIALS USING VFM**

The characterisation option of the VFM facilities was used to measure the characteristics of the cavity when a sample was loaded. The procedure followed
was a sequence of operations whereby the user graphically sees how the cavity, with material loaded, would operate over the selected frequency range. The input power is selected on the basis of the measured loss tangent of the material.\textsuperscript{3,4} The higher the loss tangent, the lower the power level selected. In the characterisation of LDPE/CF (33\%) from a frequency range of 6.5 GHz to 18 GHz, the power selected was 50 W and the amount of energy reflected varied from 0 W to 30 W ie 0\% to 60\%. In the characterisation of LDPE/GF (33\%), using the same frequency range, the input power was 125 W and the reflected energy ranged from 0W to 100W ie 0\% to 80\%. This indicated that LDPE/CF (33\%) is lossier than LDPE/GF (33\%). During characterisation of the loaded cavity, temperature variations were obtained as well as incident power and reflected power levels from the cavity containing the sample via a monitor. The incident and reflected power levels versus frequencies together with the percentage of reflectance against frequencies were monitored and recorded. From this data, the bandwidth values most suitable for microwave processing were chosen.

**Characterisation of TMC Materials from 6.5 GHz to 18 GHz.**

The total operation bandwidth for VW1500 is from 6.5 GHz to 18 GHz. For the sake of keeping the temperature low, the characterisation bandwidth was broken into four equal sections. For each quarter of the operational bandwidth of the machine, three tests were performed. This was to ensure that the tests were repeatable irrespective of the position of the specimen in the cavity. Another reason for dividing the bandwidth into four sections was because the forward
power, for a given setting, changed with respect to frequency, i.e., the amplified signal changed with respect to frequency. To minimise the error across the total operating bandwidth, the total band was therefore divided into smaller sections of roughly equal power, for a given power setting. From the data obtained it was found that the effect of position was not large and the spectra for each test were very similar.

The output power for characterisation for PS/GF (33%) and PE/GF (33%) was 125 W and the maximum temperature reached was 32°C and 33°C respectively. On the other hand, the output power for the remaining three materials were 50 W and the maximum temperature reached was 65°C. This machine does not have automatic power level control as in the case of Microcure 2100; the output power was frequency dependent. Power levelling was achieved through the change in amplifier gain with respect to frequency. As the frequency changed, the amplification factor (gain) also changed. When it increased, the forward power setting was reduced so that the output power was kept constant. When the gain decreased, the forward power setting was increased and so on. The output power for nylon 66/GF (33%), PE/CF (33%) and PS/CF (33%) in the frequency range of 9.375 GHz to 12.25 GHz was around 50 W. However, the power output was above 50 W at the lower frequency range and below 50 W at the higher frequency range. For the lower loss materials, though the output power was made at 125 W, the actual power applied on the load was less than 100 W. While analysing the characterisation results, the four quarters of the data were grouped back into one.
Considering that the reflectance is the ratio of the reflected power to the incident power, the lowest percentage of reflectance for LDPE/GF (33%) was found to be between 8.5 GHz to 9 GHz and 10.7 GHz to 12 GHz, as depicted in Figure 2. The percentage of reflectance ranged from 0 to 15. The best frequency range to process the material was therefore from 8.5 GHz to 9 GHz and from 10.7 GHz to 12 GHz in the frequency range from 6.5 GHz to 18 GHz.\textsuperscript{5} The procedure was repeated with PS/CF (33%), PS/GF (33%), LDPE/GF (33%) and Nylon 66/GF (33%) and the best frequency range to process these materials in the frequency range of 6.5 GHz to 18 GHz is tabulated in Table 1.

**Characterisation of TPC Materials from 2 GHz to 8 GHz.**

This time the Microcure 2100 was used and the frequency range of the machine was from 2 – 8 GHz. LDPE/CF (33%) was expected to have a relatively high loss tangent because of the carbon fibre contained in the matrix and thus a power level of 50 W was chosen. This was to ensure that the interaction of microwave energy and the sample was not too vigorous and that the facility could provide a complete sweep of frequency from 2 GHz to 8 GHz in a certain period of time without making the temperature in the cavity dangerously high. The temperature adjacent to the sample was monitored during the cavity characterisation process and the machine would be switched off once the temperature was over 105\textdegree C, which was not too far from the melting point of the material. Figure 3 illustrates the percentage of reflectance against frequencies. It was found that the percentage of reflectance was lowest in the frequency range of 6.5 GHz to 8 GHz. The percentage of reflectance ranged from 0 to 35. LDPE/CF (33%) was
therefore best processed in this frequency range because it absorbed a greater proportion of the incident power. The best frequency ranges to process Nylon 66/GF (33%), PS/CF (33%), LDPE/GF (33%) and PS/GF (33%) were also found to be from 6.5 GHz to 8 GHz. The percentage of reflectance of the four thermoplastic composite materials ranged from 0 to 40.

4 CONCLUSIONS

The values of the percentage of power reflectance of all tested materials in the frequency range of 2 - 8 GHz were higher than their counterparts in the frequency range of 6.5 – 18 GHz. This implies that the dielectric loss of these materials are higher at higher frequencies. In the previous study,\textsuperscript{3,4} it was found that the dielectric loss values of LDPE/GF (33%) at room temperature (24\textdegree C) were higher at higher frequencies than those at lower frequencies and the results were summarised in Table 2. By performing materials characterisation, the best range for microwave processing of a material can be discovered and followed by selective heating and processing using variable frequency microwave sources.

It is, however not possible to directly relate the power reflectance of the microwave cavity to dielectric properties of the sample within. The reason for this is that any particular frequency, a number of different modes, each having a different field pattern, will be excited within the cavity. The extent to which each one of these is excited depends on the coupling of the source to each mode, and is not susceptible to measurement. Therefore the electric field pattern within the cavity is extremely complex and unpredictable. In addition, the carbon fibres
within the sample are largely in single direction, which means that the power absorbed by the sample will depend on its orientation with respect to the filed patterns of the individual modes. Faced with such a complex situation, the best way to proceed is on a semi-empirical basis. To reduce the time consuming experimental empiricism that was required to develop relatively simple heating procedures, computer simulations of variable frequency heating may be employed.\(^5\)

**REFERENCES**


